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Comparison of two precipitation methods for the orthophosphate removal from wastewater

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ABSTRACT

The article presents a comparison of the orthophosphate removal from acid and neutral wastewaters by metal dissolution and electrocoagulation methods. The metal dissolution method is based on a similar principle to the electrocoagulation method; however, metal ions are dissolved into a solution as a result of spontaneous corrosion processes. The comparison of both methods suggests that they can provide similar results of removing phosphorus from wastewater. It was also noted that both methods introduce different amounts of iron into the solution and differ with regard to the amount of iron remaining in the wastewater. This indicates a better utilization of iron ions in the metal dissolution method and it slightly limits the problem of an excessive amount of sludge. An important parameter is the pH value in wastewater after the treatment process. Wastewater purified by the metal dissolution method was characterized by a lower increase in pH, than by the use of electrocoagulation.

Keywords: Zero-valent iron; Electrochemical treatment; Metal dissolution method; Electrocoagulation; Orthophosphates removal; Wastewater treatment

1. Introduction

Phosphorus concentrations in wastewater discharges are tightly regulated, due to concerns of the enrichment of receiving waters with nutrients and the resulting excessive growth of phytoplankton [1]. Phosphorus can be removed from wastewater biologically and/or chemically. Conventional activated sludge treatment typically reduces effluent total phosphorus concentrations to 1–2 mg P/dm^3. Enhanced biological phosphorus removal processes can lower that value to 0.1–0.2 mg P/dm^3 under ideal conditions [2]. Nevertheless, in practice, supplemental additions of chemicals such as Al or Fe(III) salts, polyaluminum chloride (PACl), and/or lime are often required to maintain acceptable effluent total phosphorus concentrations [1].

Households are responsible for more than 45% of the total phosphorus load discharged in surface water [3]. Phosphorus discharged in surface water leads to undesirable environmental problems, such as eutrophication [4]. A domestic wastewater treatment is being used as an alternative when households are located far from an existing sewer network. Stimulated by the European legislation, decentralized wastewater treatment systems (i.e. with a population equivalent smaller than 10) are being employed with increased

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frequency. Implementing phosphorus removal in decentralized wastewater treatment is becoming a necessity. Decentralized systems should be robust, simple, and should not require too much or too a complicated maintenance. Important features for the selection of a phosphorus removal strategy comprise the impact on the operation and maintenance, the total cost of implementation and maintenance, the impact on the sludge production (and handling) and the dimensions [5].

The conventional biological phosphorus removal process is not the most suitable technique for decentralized wastewater treatment. A significant process control (of which the system owner cannot take care themself) is necessary. Similarly, phosphorus removal through the dosing of chemicals such as aluminum or iron salts cannot be applied. Almost no extra space is required for the implementation of this technique, but the maintenance and the operational costs are significant. Sludge production will increase significantly resulting in the need for more frequent maintenance. The result is an increase in the yearly operating and maintenance costs, as sludge removal will have to be carried out more frequently. The chemicals are expensive and their handling and storage make additional cost. Finally, this technique can only be efficient when an online total phosphorus measuring device is installed, due to the varying characteristics of domestic wastewater, phosphorus concentration, and required effluent values [5]. Unquestionably, effective methods of phosphorus compound removal include precipitation methods. The most common is the chemical precipitation method, based on applying Fe, Al, or Ca salt to the solution. The phosphorus removal efficiency can even 90%, however this is dependent on many factors (e.g. coagulant doses, pH, initial phosphate concentration or coagulant type) [6]. Electrocoagulation is another way of introducing metal ions. It is based on applying metal ions by use of electrolyte dissolving of suitable electrodes. Primarily iron and aluminum electrodes are used. The efficiency of phosphorus removal can be almost 100%. However, it depends on many factors (the electrolysis time, current density, pH, initial concentrations of P and electrode type) [7,8]. During the phosphorus removal processes, both the conventional precipitation method and the electrocoagulation produced a large amount of wastewater sludge. Previous research shows that electrocoagulation is a low sludge producing technique [9]. The electrocoagulation process avoids uses of chemicals, and so no possibility of secondary contamination (e.g. Cl^-, SO_4^{2-}) caused by chemical substances added at high concentration as when chemical coagulation of wastewater is used [9]. However, the flocs in electrocoagulation-treated suspensions had a higher density and tended to be larger than flocs formed in suspensions dosed with iron chloride or iron sulfate. The kinetics of settling and filtration of electrocoagulation-treated suspensions were accelerated, demonstrating the interest of electrocoagulation as an alternative to chemical conditioning [10]. Another disadvantage is that high conductivity of the wastewater suspension is required and the need to use electricity which may be expensive in many places [8].

Based on electrocoagulation is also the method of metal dissolution proposed by the authors. The main difference between the aforementioned methods lies in the way of applying the precipitation agent to the solution. The metal dissolution method is based on similar premises as the electrocoagulation method, however, metal ions here are dissolved into the solution as a result of spontaneous corrosion processes (no need to use electricity) and, due to their further transformations in the wastewater environment, they are responsible for phosphorus removal. As a result of exposing the steel to the wastewater environment, heavy corrosion is observed.

Chemical reactions [11,12]:

$$\text{Anode:} \quad Fe_{em} \rightarrow Fe^{2+} + 2e \tag{1}$$

$$\text{Cathode:} \quad 2H^+_{sol} + 2_e \rightarrow 2H_a \quad \text{for} \quad pH > 7 \tag{2}$$

$$2H^+_{sol} + 2_e \rightarrow H_{2g} \quad \text{for} \quad pH < 7 \tag{3}$$

OH^- anions (formed as a result of water dissociation, being electricity carriers in the water together with H^+) flow to the anode and combine with bivalent Fe^{2+} ions forming hardly soluble $Fe(OH)_2$ ferrous hydroxide [12]. Apart from forming hydroxides, other compounds of Fe^{2+} (such as $Fe_3(PO_4)_2$) form in the wastewater. At the anode, OH^- might further transfer into H_2O and O_2, and then Fe^{2+} can be oxidized to Fe^{3+}, then precipitate in the form of Fe^{3+}. Similarly, as in the process of coagulation, the compounds (Fe^{2+}, Fe^{3+}) cause precipitation of phosphorus compounds as a result of ongoing physicochemical reactions. The metal dissolution method generates significantly less wastewater sludge than the electrocoagulation method [10]. In addition, as the electrocoagulation method, this process avoids the use of chemicals, and so no possibility of secondary contamination (e.g. Cl^-, SO_4^{2-}) caused by chemical substances added at high concentration, as can occur when chemical coagulation of wastewater is performed [13].

The following article presents a comparison of the effects of orthophosphate removal from wastewater with an acid reaction by two methods: the metal dissolution method and the electrocoagulation method.

2. Methods

2.1. Structure of the examination post

The examination has been carried out in a static system at room temperature (20°C). A glass vessel with the capacity of max 2.5 dm^3 was a single element of the research station. A filler was placed in the vessel where wastewater was also added (2.2 dm^3). The filler was made of 10 electrodes (250 mm × 50 mm × 2 mm) made from two types of steel (5 from black steel and 5 from stainless steel—Table 1). Prior to use, the electrodes were exposed for 2 weeks to a wastewater environment for activation (in order to achieve the products of corrosion—iron oxides—on the surface of the electrodes). The activation of electrodes ensures a more efficient course of the wastewater treatment processes [14]. The distance between the electrodes was 5 mm. The total surface of immersed parts of the electrodes was 141,400 mm^2 per station (14,140 mm^2 per one electrode).

The research stations were divided into two groups. The first group included three glass vessels in which the electrodes were bridged with a copper wire to ensure the flow of electrons between the anode and the cathode (Fig. 1)—the metal dissolution method. The second group included the other three glass vessels in which each electrode was incorporated into an electric circuit of direct current of 0.1 A (Fig. 1)—the electrocoagulation method. In both cases, electrodes made of black steel served as anodes. Each station included a glass vessel with a magnetic stirrer type MS 11HS by WIGO company with the stirring element in the form of a cylinder (ϕ 0.7 cm and length of 5 cm). The velocity of the stirrer was 1,500 rpm.

2.2. Type of wastewater used

The experiment was conducted with synthetic wastewater of pH 5 and pH 7 (to represent wastewater of different corrosivity) [7]. The concentration of orthophosphates reached ca. 13–16 mg P/dm^3 (average concentration of phosphorus in municipal wastewater) [15]. The synthetic wastewater used in the experiment was prepared by dissolving dipotassium phosphate (K_2HPO_4) in tap water. The pH value of the wastewater was controlled using potassium hydroxide solution (KOH—5 M) and nitric V acid (HNO_3—5 M). The tap water was supplemented only with K_2HPO_4, KOH, and HNO_3.

2.3. Analytical methods

Samples of wastewater were collected directly from the research station after decanting. Afterward they were filtered by a filter with a pore diameter of 0.45 μm. The content of orthophosphates and total iron in the samples examined was determined with the spectrophotometric method (spectrophotometer HACH DR/2010) following measurement procedures provided by the manufacturer. The analysis of the phosphate concentration was carried out using the spectrophotometric ascorbic acid method (PhosVer method no. 8048—wavelength 890 nm) and the iron concentration using the phenanthroline method (FerroVer method no. 8008—wavelength 510 nm). The measured concentrations of the parameters studied were characterized with the use of arithmetic mean and standard deviations. The advanced hypotheses, aimed at evaluating differences between particular elements, were verified with the Student's *t*-test and analysis of variance. The differences were found to be significant at $p < 0.05$.

3. Results and discussion

The physicochemical methods of removing phosphorus compounds from wastewater, both the widely applied method of phosphorus precipitation with metal salts, the method of electrocoagulation, and metal dissolution method are highly effective ones [16–19]. Wastewater treated by those methods most often guarantee to achieve phosphorus concentration levels required by law for both municipal and industrial wastewaters [20]. Electrocoagulation is already widely used, not only in phosphorus compounds removal processes [21], and creates a significant total

Table 1
Elements composition of electrodes

	Content (%)								
Type of steel/Type of electrode	C	Mn	P	Al	N	S	Si	Cr	Ni
Black steel/Anode	0.130	0.52	0.011	0.043	0.006	0.005	–	–	–
Stainless steel/Cathode	0.024	1.64	0.027	–	0.049	0.002	0.39	10.1	8.1

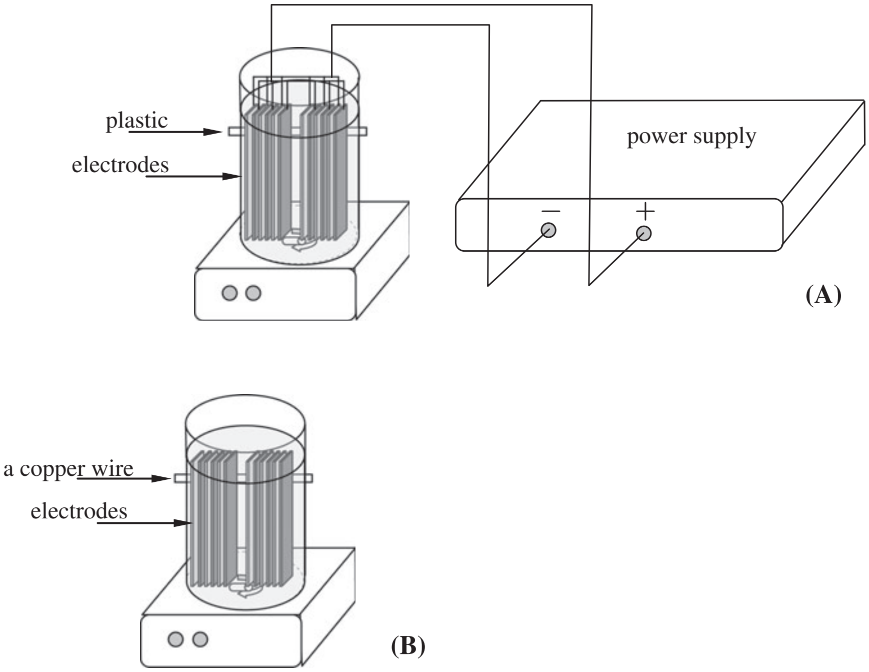

Fig. 1. Scheme of a research station (A) the electrocoagulation method and (B) the metal dissolution method.

phosphorus reduction even above 80% [22]. The reduction is similar, by the metal dissolution method. The phosphorus concentration in wastewater using this method decreases below 2 mg P/dm^3, and often below 1 mg P/dm^3 and the efficiency exceeding 80% [17,18]. Even better results observed in the case of orthophosphate removal, where efficiency is increased up to 100% and the concentration can reach a level below 0.1 mg of P-PO_4^{3-}/dm^3 [17,18]. Everything, of course, depends on the process conditions and the wastewater type. The methods utilize low solubility of metal phosphates and their sorption on the surfaces of the forming agglomerates. The mechanism is complicated and consists of transforming phosphorus compounds into insoluble forms, and then separating them from the wastewater by flotation, sedimentation, or filtration. The main difference between the methods lies in the way of applying the precipitation agent.

The study presents evaluation of the effectiveness of orthophosphates removal from two types of synthetic wastewater with two methods: metal dissolution method and electrocoagulation. The study was carried out for two types of synthetic wastewater differing in pH.

pH is an important factor in removing contaminants from wastewater, especially in the case of the metal dissolution method. In this method, the amount of metal present in the wastewater is doubtlessly a function of a speed of the corrosion processes. The more effective corrosion process is, the larger the amount of ions gets into the solution. It can therefore be supposed that corrosion processes will be inhibited in wastewater of about pH 7, but in the whole range of pH from 0 to 14 the Fe/Fe^{2+} potential is lower than the hydrogen electrode potential. It is therefore possible that iron dissolves on the anode in the whole range of pH, but the process will not run very fast. Probably because of this, decrease in orthophosphate concentrations in the treated wastewater with the passage of time is minimal—after 2,680 min of the process efficiency was 56% (Fig. 2), and total iron concentration is low (max 0.94 mg Fe/dm^3) (Fig. 3). In the case of the metal dissolution method in the wastewater of pH 7, the amount of the iron ions dissolved into the solution may be influenced by the increase in the surface of the filler or by increase in the contact time [17].

The amount of metal ions present in the solution in electrocoagulation determines the value of the current applied to the electrode. In this experiment, a direct current of 0.1 A per station was used. This was sufficient to achieve complete removal of orthophosphates from the wastewater after 45 min (Fig. 2). Total iron concentration, not used in the process of removing orthophosphates, was also higher initially than in the metal dissolution method. When the neutral wastewater (about pH 7) was purified, the best method is probably electrocoagulation. Using the metal dissolution method would require longer

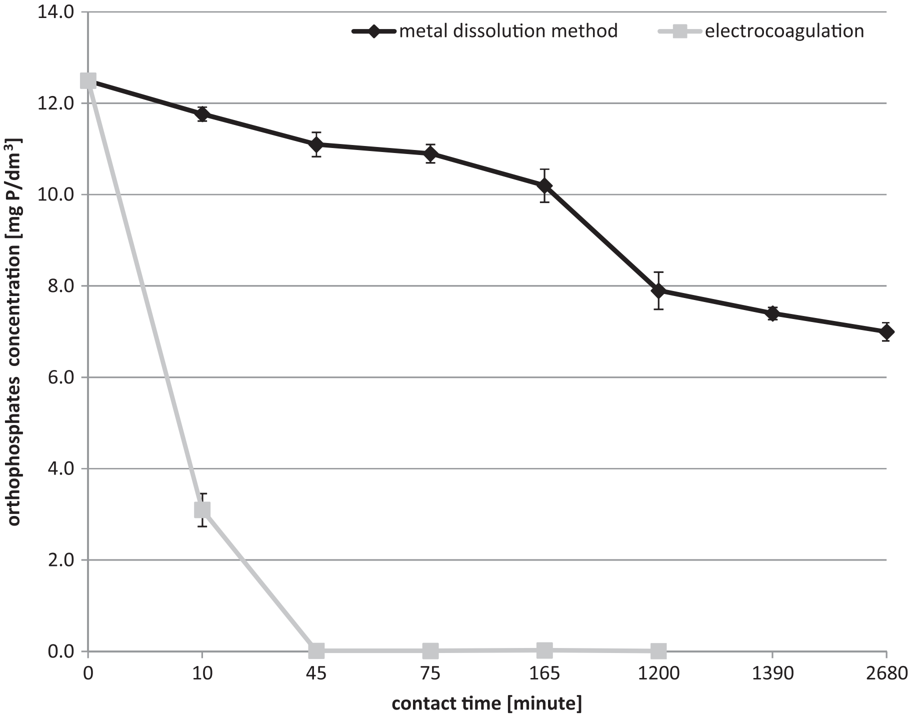

Fig. 2. Orthophosphate concentration in wastewater (pH 7) treated with the metal dissolution method and the electrocoagulation method.

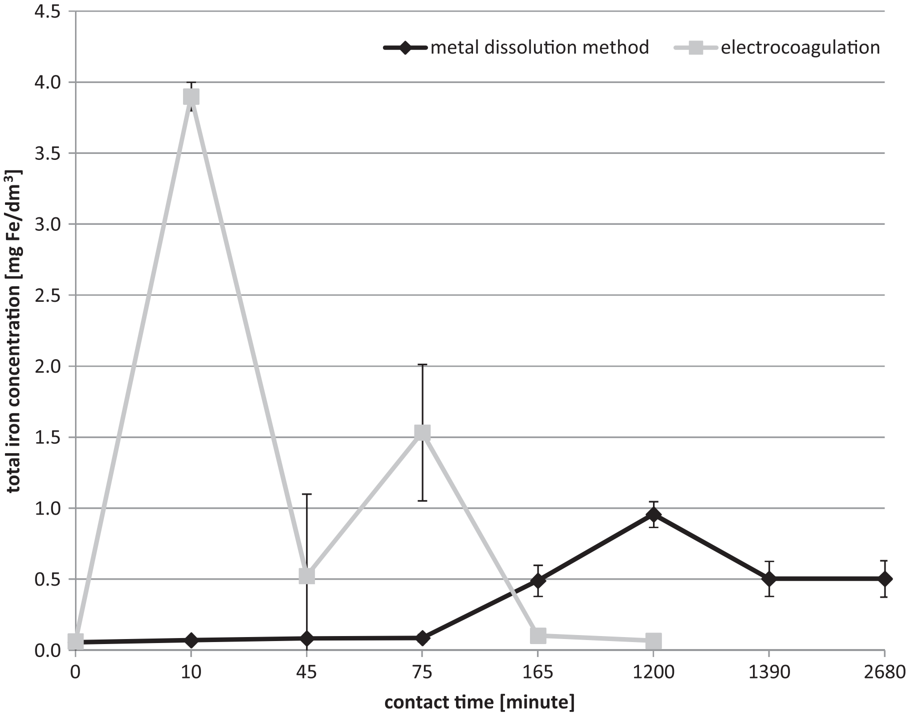

Fig. 3. Total iron concentration in wastewater (pH 7) treated with the metal dissolution method and the electrocoagulation method.

contact time, or a larger contact surface. Another way of increasing the amount of iron ions in the wastewater with metal dissolution method is a pH decrease. The decrease to pH 5 greatly improves the orthophosphates removal (Fig. 4) and increase in iron concentration (Fig. 5). Also, in such a case, orthophosphates are removed much faster when using electrocoagulation, than using the metal dissolution method. However, the metal dissolution method can achieve satisfactory results—reducing the concentration to 99.7% (after 45 h and 25 min contact with the filling) (Fig. 4). The contact time is much longer, but does not require the use of an electric current (generating additional costs). As already mentioned, the contact time can be reduced by increasing the contact area filled with wastewater [18]. Depending on the wastewater type, it is worth considering the possibility of using the metal dissolution method. It does not require the application of an electric current, which is a drawback of electrocoagulation, but it requires a longer contact time (or larger contact surface).

Unfortunately, dosing iron ions to wastewater to remove phosphorus causes secondary contaminations with those ions. The iron (II) presence in the treated wastewater has a negative impact on the nitrification process (1 mg Fe^{2+}−0.29 mg O_2 consumed during the oxidation process to Fe^{3+}). On the other hand, it supports the reduction process of nitrite to nitrogen gas [23]. If the excess iron penetrates into the aquatic environment, it may adversely affect the reservoir. High iron concentrations cause disturbances in the growth of plants and the concentration of 1–2 mg/dm^3 is toxic to fish. Therefore, it is important to control this water pollution index.

Some iron ions (during the phosphorus removal process) remain unused. This is a problem to contend with for electrocoagulation supporters as well as for the ones in favor of the iron dissolution method [17,18,24,25]. According to the literature, the iron concentration (at points of maximum process efficiency) even reach 40–60 mg Fe/dm^3 during electrocoagulation [26], 140–160 mg Fe/dm^3 during conventional coagulation ($FeCl_3$) [26], and 2.0–13. 5 mg Fe/dm^3 during the metal dissolution method [27]. In the case of electrocoagulation, iron ions are much better utilized than in the case of a conventional coagulation [16]. Sufficiently long process duration will result in a gradual decrease in the iron concentration [26]. This is not always to a satisfactory level as concentration does not always decrease below the recommended standard −10 mg Fe/dm^3 [20]. In this case, as suggested by Wysocka and Kościelniak [27], other methods should be considered e.g. the oxygenation process in order to eliminate secondary contamination.

Each of those methods introduces the metal into the solution in a slightly different manner. Owing to that fact, controlling of that process should be performed a bit differently. The amount of the metal present in the wastewater after the processes of phosphorus compound removal with the dissolution method is undoubtedly a function of the speed of corrosion processes, the surface of the filler, and the wastewater-filler contact time [17,18]. In the case of electrocoagulation, it is a function current density and

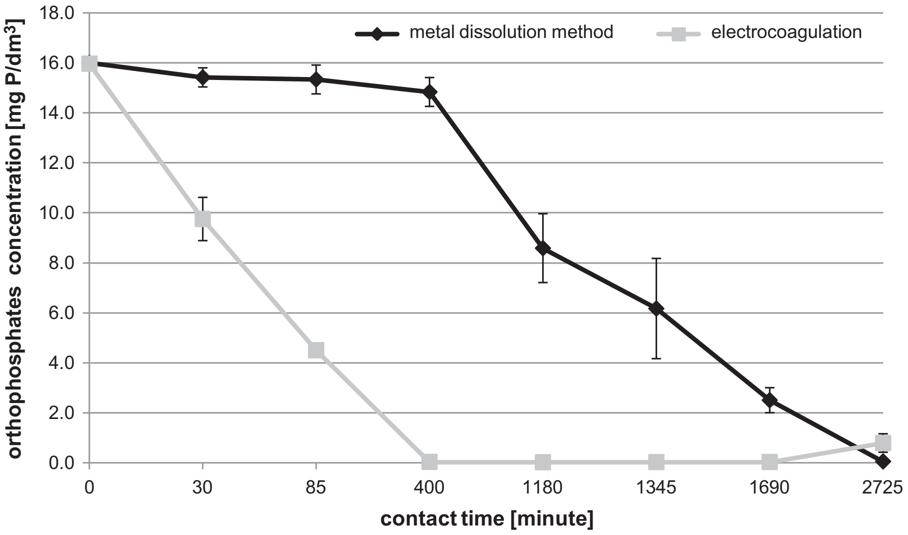

Fig. 4. Orthophosphate concentration in wastewater (pH 5) treated with the metal dissolution method and the electrocoagulation method.

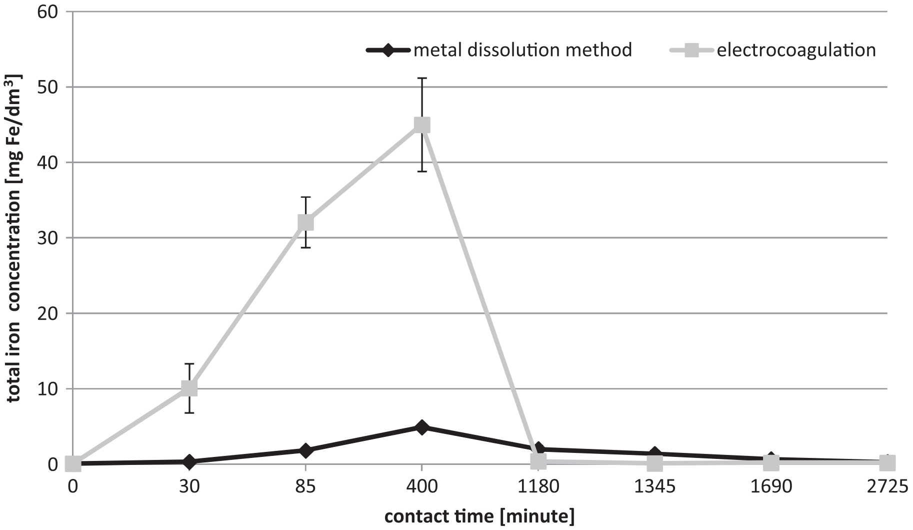

Fig. 5. Total iron concentration in wastewater (pH 5) treated with the metal dissolution method and the electrocoagulation method.

the wastewater-filler contact time [8,28]. Finding the optimal solution i.e. ensuring a proper reduction of phosphorus compound concentration and the lowest values for the iron ions concentration (the secondary contamination) is necessary to secure a correct course of the treatment process. Thus, controlling the advances of the filler corrosion (metal dissolution method) and current density (electrocoagulation) becomes indispensable, so that excessive liberation of iron ions into the solution could be avoided.

However, the amount of the secondary wastewater contamination depends not only on the speed of iron dissolution, but also on the parameters of treated wastewater. In the treated wastewater, compounds of iron may occur both in a dissolved and a colloidal form. The higher the solubility of the iron compounds the greater the amount of ions remaining in the solution. Also the solubility is decided not only by such parameters as the reaction or temperature of the process, but also by the coexistence of other ions in the solution. Wastewater with pH 7 purified by the metal dissolution method did not show very high levels of "unused" iron (Fig. 3), which is the result of secondary contamination (max 0.95 mg Fe/dm^3). Much higher concentrations have been reported in wastewater treatment by electrocoagulation (max 3.9 mg Fe/dm^3) (Fig. 3). Much higher concentrations of "unused" iron appear to be at lower pH 5 pH. During the process of the electrocoagulation, highest recorded value was 45 mg Fe/dm^3, and during the application of the metal dissolution method is only 4 mg Fe/dm^3 (Fig. 5).

Despite the low iron concentration, the phosphorus removal efficiency by the metal dissolution method at pH 5 was substantial. pH 5 is a corrosive environment for the iron electrodes. Phosphate removal takes place by the formation of $FePO_4$ and $Fe(OH)_3$, although the $FePO_4$ is favored in relatively low pH values (pH 5) [7]. Solubility of the $FePO_4$ increased with increasing pH [7]. $FePO_4$ has the minimum solubility within this pH range [7]. Therefore, $FePO_4$ formation removes phosphates without increasing secondary iron concentration. In the case of electrocoagulation, the metal dissolution rate is much greater than in the metal dissolution method. Near the electrode, a significant amount of iron ions are released which will have limiting effect of PO_4^{3-} ion availability. This favors the iron hydroxides formation, which will raise the pH, and will increase the concentration of dissolved and undissolved iron forms. The methods differ with regard to the amount of iron remaining in the wastewater (also in a suspended form). This indicates a better use of iron ions in the metal dissolution method, and it slightly limits the problem of an excessive amount of sludge.

An important parameter is the pH value in wastewater after the treatment process. The use of electrocoagulation (steel electrode) changes the pH of wastewater to above pH 10 [8]. During the test with the use of pH 5 wastewater and pH 7 wastewater also achieved such values (Figs. 6 and 7).

But if the process was stopped at the right moment (400-min wastewater with pH 5 (Fig. 6) and 45-min wastewater with a pH 7 (Fig. 7)), the pH value

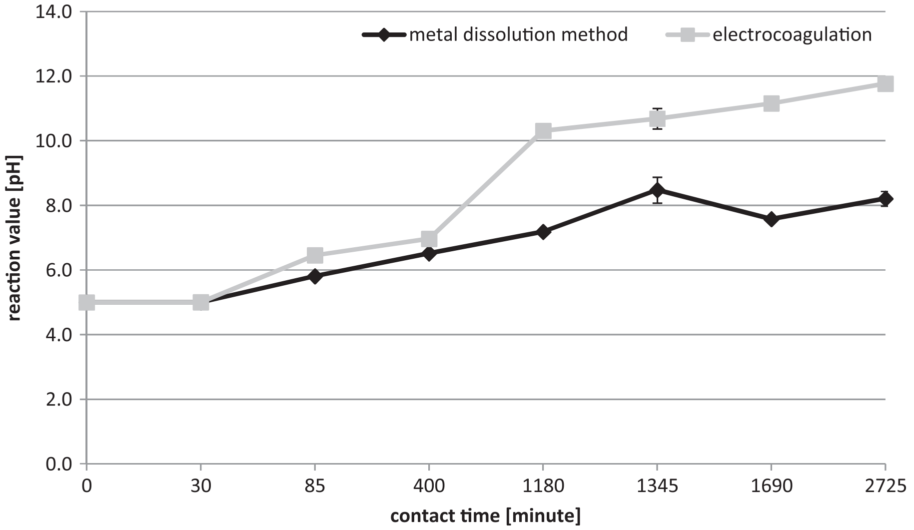

Fig. 6. The pH value in wastewater (pH 5) treated with the metal dissolution method and the electrocoagulation method.

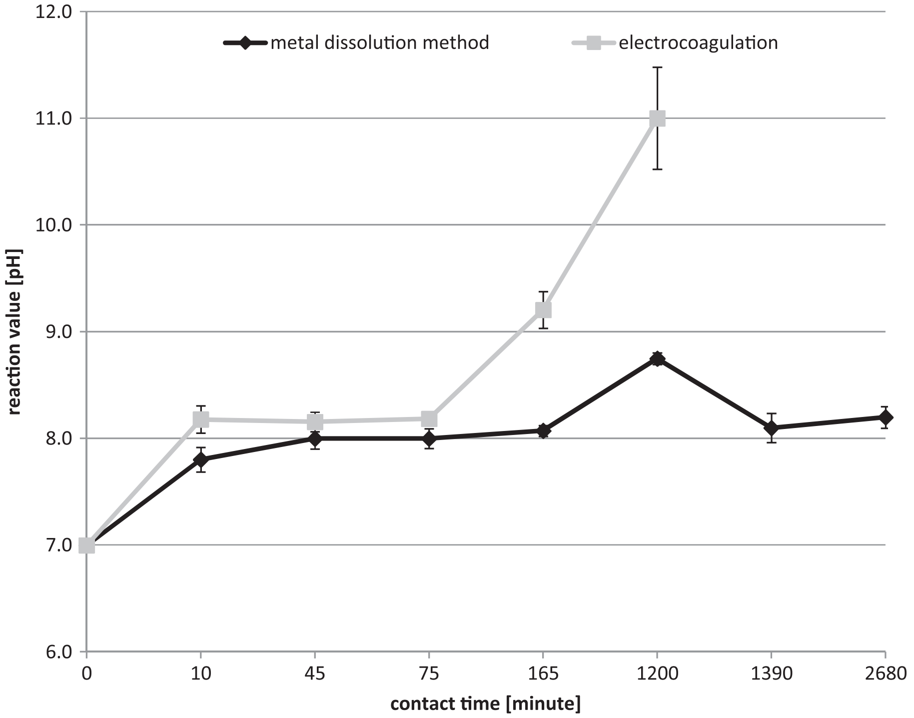

Fig. 7. The pH value in wastewater (pH 7) treated with the metal dissolution method and the electrocoagulation method.

fluctuated at around 8. It therefore strictly observes the process conditions. In the case of the metal dissolution method, the reaction wastewater fluctuated at around pH 8 but the process was stopped after reaching a satisfactory orthophosphate reduction (Figs. 6 and 7).

The process should be stopped when reaching satisfactory phosphorus removal result, when the pH of the effluent meets standards and reaching satisfactory phosphorus removal results.

Methods although very similar, differ in the way of conducting the process and the quality of treated wastewater. Economic calculations should be performed for particular wastewater types, so to select the most optimal wastewater treatment method.

4. Conclusions

Wastewater treatment by the metal dissolution method can compete with the electrocoagulation method. This is dependent on the kind of applied wastewater. The process speed in wastewater of pH 7 was very small and the method application would require much longer contact times or a significant increase in the filling surface. In the case of acidic wastewater (pH 5), orthophosphates removal efficiency is high achieving practically complete removal. However, this is a method that requires a slightly longer retention time (wastewater in contact with filling) than the electrocoagulation method but without requiring the use of a current. After the treatment process, like with the other precipitation methods, there is secondary iron contamination of wastewater (slightly lower for the metal dissolution method). Electrocoagulation and metal dissolution methods cause an increase pH, bringing it to a value of pH 8, but if the orthophosphates removal process is complete, continuation of the electrocoagulation causes an increase to about pH 12. It is not without significance to continue wastewater treatment processes and for the environment to which the treated wastewater is placed.

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